A Simple Device for Constant Temperature Control in a High Pressure Girdle Apparatus

KARL-AXEL WILHELMI and WALTER BURGER

Institute of Inorganic and Physical Chemistry, University of Stockholm, Box 6801, S-113 86 Stockholm, Sweden

A high-pressure girdle apparatus for work up to 60 kb and 1700°C is described. A new method is used to introduce the thermocouples. Good constant temperature control is maintained by a simple device.

A convenient way of measuring and controlling the temperature in an internally heated high pressure apparatus is to control the input of the electrical power. The temperature scale is then established with the aid of the pressure dependence of certain known melting points.

The most direct method for measuring the temperature is to use thermocouples within the pressurized zone. However, the thermal EMF is influenced by pressure and the pressure dependence is a correction term which must be taken into account. The magnitude of the correction term to be added to the thermocouple reading depends, however, on the way the wires are introduced into the pressure region. The stretching and squeezing to which the thermocouple wires are subjected when a high pressure cell is loaded often introduces additional difficulties.

The purpose of this article is to give a short presentation of the girdle apparatus used at this Institute and to describe the work which has been made to overcome the difficulties mentioned above. A new method has been employed for introducing the thermocouple into the high pressure chamber and a simple device has been constructed to obtain constant temperature control in the instrument.

THE HIGH PRESSURE EQUIPMENT

Various features of the Bridgman anvil device and the piston-cylinder apparatus of Boyd and England are combined in the "Belt" designed by Hall.[1] Several modifications of this apparatus, with somewhat simplified geometries, have been developed during recent years. The equipment used at this

Institute is in some respects an improved version of the apparatus introduced some years ago by Daniels and Jones.[2]

The high pressure girdle apparatus consists of two tungsten carbide pistons and a carbide vessel (quality S10) supported radially by steel cylinders (Coromant, Sandviken). Pressure is generated by advancing the pistons with a hydraulic press—maximum ram load 500 tons. The gasket seals, which are partly deformed while compressing the pistons, consist of glass fibre reinforced teflon. The pressure transmitting medium has so far been pyrophyllite.

Internal heating is provided by passing an electric current from an AC power transformer *via* the pistons through the 0.1 mm thick platinum tube (outside diameter 3 mm) situated in the middle of the girdle. The electrical heating effect can be increased to a maximum of 400 A and 1 V across the specimen tube.

The tilting of the girdle assembly which occasionally occurs during the pressure cycle, due to asymmetries in the gasket function, has been avoided in the following way. The girdle is maintained in a horizontal position by means of eight heavy springs, placed under and above it and electrically isolated by PVC knobs from the cradle which supports the high pressure assembly. The upper piston is attached to the upper part of the cradle. To facilitate the alignment of all parts, the plate holding the lower piston and girdle assembly is placed on tracks and can be positioned by a pre-set stopping pin so that the center of the upper piston is directly above the lower piston. The upper parts of the cradle and the upper piston can be lifted by two different pneumatic pistons.

The ram load is measured with the aid of pressductor equipment (ASEA) situated between the high pressure apparatus and the upper yoke of the press. The pressure calibration of the apparatus is made by the usual procedure of observing the electrical resistance change in a Bi wire situated inside a silver chloride sleeve which replaces the platinum tube.

CONSTANT TEMPERATURE CONTROL DEVICE

The regulating voltage for temperature control is taken across a Wheatstone bridge containing the platinum tube ($R_1 \approx 5 \times 10^{-3}$ Ω at ambient temperature) as indicated in Fig. 1. In this way the regulation is based only on the temperature coefficient of the resistivity for platinum. The value of this coefficient α_{Pt} is $\sim$0.003 (°C)$^{-1}$ which means that the resistance changes 0.3 % for a change in temperature of 1°C.

R_4 is a constant (10 Ω) resistance, R_2 a ten-turn potentiometer ($R_{2\ max} = R_4$). R_3 consists of a variable length of the water-cooled brass tube which gives an adjustable resistance independent of the current ($R_3 \approx R_1$).

At a pressure of 30 kb and a temperature change from 500°C to about 1500°C, the required heating voltage increases by approximately 100 %. A 1°C deviation gives a voltage change of the order of 1 mV.

After amplification and rectifying, this current then passes through the temperature regulator (Reg. in Fig. 1). The potentiometer R_2 can be calibrated

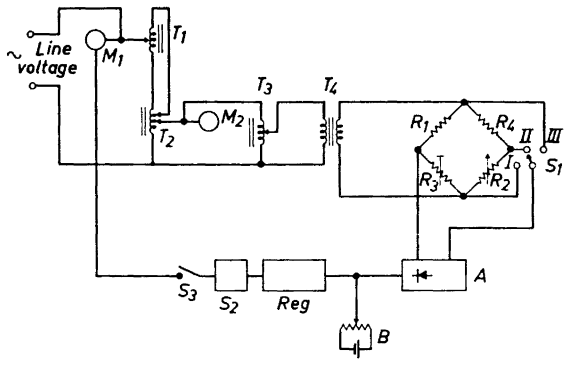

Fig. 1. Block diagram of constant temperature control device.

M_1 servomotor; relay operated by S_2.
M_2 servomotor.
T_1 variable transformer; operated by M_1.
T_2 main transformer for adjusting the avialable temperature by the manually switched motor M_2.
T_3 30 amps auto plug-transformer.
T_4 power transformer supplying 2 volts and 400 amps.
R_1 resistor, representing the specimen holder.
R_2 ten-turn potentiometer.
R_3 temperature stabilized resistor.
R_4 bridge resistor.
S_1 switch for coupling bridge (II), or for voltage measurements (I, III).
S_2 regulator relay to switch M_1 in left, right or at rest position.
S_3 on-off switch.
A AC-amplifier and rectifier.
B compensation voltage.
Reg temperature regulator.

in a temperature scale and therefore forms an arrangement for approximate temperature adjustment.

Before heating the Pt-tube, S_1 is in the position II and R_2 is adjusted to its maximum position where $R_2=R_4$. The bridge is then balanced at ambient temperature by varying R_3 while keeping the supply voltage so low that no heat is produced. The fine regulation transformer T_1 is adjusted to its middle position while the main transformer T_2 is adjusted to a position approximately corresponding to the desired temperature. S_3 is in the off position.

The temperature then rises as indicated by the thermocouple galvanometer (see below). Now the bridge can be balanced at any temperature by the potentiometer R_2. When the desired temperature has been reached (after 20 min), S_3 is switched on and R_2 is turned slightly off balance so that a too high temperature makes M_1 decrease the heating voltage and *vice versa*.

In order to drive the regulating motor in the correct direction when required, the temperature regulator must be of a two-point type (Getrosist 606B 44).

The compensation voltage B is used for zero point adjustment.

MEASUREMENT OF SPECIMEN TEMPERATURE

The temperature measurement is made using the thermocouple principle. In order to avoid having to lead thermocouple wires through the teflon gasket, a thermocouple arrangement has been built into the pressure chamber and consists of the platinum tube mentioned above and a short PtRh (10 %) wire pricked through the Pt tube and led through a small hole in the pyrophyllite body. The wire is bent at right angles inside the tube (see Fig. 2).

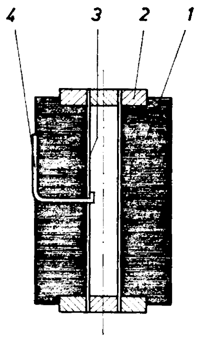

Fig. 2. Cross section of pyrophyllite transmission cylinder; 1) pyrophyllite, 2) brass contact, 3) platinum tube, 4) PtRh (10 %) wire.

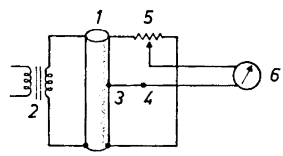

Fig. 3. Electrical circuit for the EMF measurement: 1) platinum tube, 2) power transformer, 3) hot junction, 4) reference junction, 5) AC zero potentiometer, 6) EMF.

In this way, mechanical damage to the tube, which could affect the heat distribution, is prevented as far as possible. The point of contact represents the hot point of the thermocouple. The specimen is then tightly packed into the tube. The cold point of the Pt—PtRh thermocouple is on the outside of the pyrophyllite body in contact with the water-cooled tungsten carbide belt.

Fig. 3 shows the electrical circuit for the EMF measurement. The potentiometer is an integral part of an AC bridge; it is adjusted until AC fluctuation on the temperature galvanometer disappears.

A check of the thermocouple method has been performed by inserting a second thermocouple into the middle of the specimen (for this purpose Al_2O_3 powder was used) using a similar experimental arrangement at atmospheric pressure. This EMF was in exact agreement with the EMF determined as described above.

For phase studies, the cooling time of the sample is of great importance. When the current is cut off, the specimen temperature decreases from 1600°C to approx. 200°C in a few seconds. By using a gear reduction set together with the servometer M 1, this time can, however, be extended to several days.

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